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## PATENT SPECIFICATION

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### COMPLETE SPECIFICATION

#### Recovery of Phosgene

We, FARBENFABRIKEN BAYER AKTIEN-GESELLSCHAFT, of Leverkusen-Bayerwerk, Germany, a Body Corporate, organised under the Laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to a process for the recovery of phosgene from waste gases containing phosgene and hydrogen chloride.

15 In the production of isocyanates by treating amines with phosgene, a large excess of phosgene is required. The economy of this process, therefore, depends on the recovery of substantial quantities of excess phosgene, which is obtained in admixture with hydrogen chloride formed in the reaction.

20 It has been proposed to separate phosgene from hydrogen chloride by washing the waste gases from this process with cold *o*-dichlorobenzene. This process is carried out on a large scale by circulating the *o*-dichlorobenzene, which is first cooled in a brine cooler, then passed over a washer where it is brought into contact with the waste gases containing phosgene, heated to 60–70° C. in order to remove the absorbed phosgene, and then returned to the cycle. (See BIOS—Miscellaneous Report No. 1; The German Plastics Industry, Appendix 8, pages 6 and 14). This process, however, has serious drawbacks which cause considerable losses of phosgene. Not only are large quantities of phosgene carried away together with the hydrogen chloride escaping from the washer, but also the cold *o*-dichlorobenzene, especially the *o*-dichlorobenzene containing phosgene, absorbs substantial quantities of hydrogen chloride, which greatly contaminate the recovered phosgene. Another disadvantage associated with this prior art method is that the recovered phosgene is in the form of gas and must be used at once since it cannot be stored as a gas.

45 The present invention provides a very simple and economic process for the recovery of phosgene from waste gases containing phos-

gene. Other advantages of the process are set out below.

50 In accordance with the present invention, we have found that phosgene can be recovered from waste gases containing phosgene and hydrogen chloride by passing the waste gases upwardly through a cooling apparatus which cools the gases to a temperature below the boiling point of phosgene and above the boiling point of hydrogen chloride, down which cooling apparatus liquid phosgene condensed from the waste gases by the cooling trickles 60 under gravity.

65 Preferably the gases are cooled in the cooling apparatus to a temperature of –40 to –60° C. The gas mixture of hydrogen chloride and phosgene entering the cooling apparatus is preferably at a temperature below the boiling point of phosgene (8° C.), i.e. 0 to 5° C., and is further cooled by contact with the condensed liquid phosgene which trickles down the cooling apparatus under gravity. 70 Liquid phosgene issues through the bottom of the apparatus and gaseous hydrogen chloride escapes at the top. The liquid phosgene obtained by the herein-described recovery process contains up to 0.7% of hydrogen chloride and the escaping gaseous hydrogen chloride still contains some phosgene, the quantity depending upon the partial pressure at the temperature of the escaping residual gas.

75 The gas mixture of hydrogen chloride and phosgene is preferably cooled in a tubular cooler, the tubular parts of which are cooled by an appropriate cooling medium, for instance boiling difluorodichloromethane or difluoromonochloromethane. 80

85 The invention is further illustrated by way of example in the accompanying diagrammatic drawings, which show sectional views of two apparatus wherein phosgene can be recovered from waste gases containing phosgene by the herein-described process.

90 Referring first to Fig. 1, the waste gases enter the cooler 2 from below through the pipe 1 and pass upwardly through the cooler where they come into contact with condensed liquid phosgene which trickles downwards

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under gravity and discharges into the storage tank 4. The non-condensed gases escaping from the cooler 2 through the pipe 5 consist substantially of hydrogen chloride which is eliminated in the washer 6 by irrigation with water fed through the pipe 8 and discharged as an aqueous solution through the pipe 7. Any inert gases which may be present are exhausted through the pipe 9 by the suction fan 10, which is arranged at the end of the apparatus and maintains a slight sub-atmospheric pressure within the whole apparatus, and forced into a chimney. The phosgene, which is obtained in the liquid state, can be taken from the storage tank 4 whenever it is desired.

The amount of phosgene which is recovered by the hereindescribed process is substantially larger than that obtained according to the above-mentioned prior art process. It is a surprising feature of the present invention that the recovered phosgene contains only 0.5—0.7% of hydrogen chloride, which allows the phosgene to be used for reactions with amines without purification.

In the process for the recovery of phosgene just described, difficulties may arise when the apparatus is leaky. Moisture will then be sucked into the apparatus together with air and, in admixture with the hydrogen chloride, will cause strong corrosion. Not only may such corrosion endanger the operation of the apparatus, but the recovered phosgene is contaminated with metal chlorides, especially iron chloride, which limit its usefulness since metal chlorides cause inconveniences when the recovered phosgene is again used to treat amines.

It is within the scope of the invention to obviate these difficulties by maintaining a slight superatmospheric pressure in the cooler. This is accomplished, as shown in Fig. 2, by arranging a preferably automatic, high pressure relief valve 11 behind the cooler 2 into which the waste gases are led through the pipe 1 and from which the liquid phosgene flows into the tank 4. A pressure of 100—200 mm of water is maintained in the cooler by means of this high-pressure relief valve 11. By this arrangement any moisture is safely prevented from entering the cooler and the recovered phosgene is water-clear. Any leakages are

immediately perceived due to the phosgene odour, and can be remedied. The residual gases, as in the case of Fig. 1, are passed through the pipe 5 into the washer 6 where the hydrogen chloride is eliminated with water admitted through the pipe 8. The hydrogen chloride in the form of an aqueous solution, is discharged through the pipe 7. The washer 6 is connected with the suction fan 10 by the pipe 9. The washer 6 connected at the outlet side of the recovery apparatus is preferably maintained at the sub-atmospheric pressure of the suction fan 10 arranged at the end of the recovery apparatus. In this way a sub-atmospheric pressure may be temporarily produced and maintained in the recovery apparatus by means of a by-pass pipe 12 which by-passes the high pressure relief valve 11, should a sub-atmospheric pressure be required for any reason.

What we claim is:—

1. Process for the recovery of phosgene from waste gases containing phosgene and hydrogen chloride, which comprises passing the waste gases upwardly through a cooling apparatus which cools the gases to a temperature below the boiling point of phosgene and above the boiling point of hydrogen chloride, down which cooling apparatus liquid phosgene condensed from the waste gases by the cooling trickles under gravity.

2. Process as claimed in claim 1, wherein the cooling is carried out at a pressure slightly above atmospheric.

3. Process as claimed in claim 1 or 2, wherein the waste gases are cooled in the apparatus to  $-40$  to  $-60^{\circ}$  C.

4. Process as claimed in any of claims 1 to 3, wherein the waste gases entering the cooling apparatus are at a temperature of  $0$  to  $5^{\circ}$  C.

5. Process for the recovery of phosgene from waste gases containing phosgene and hydrogen chloride substantially as described with reference to Fig. 1 or Fig. 2 of the accompanying drawings.

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FIG.1

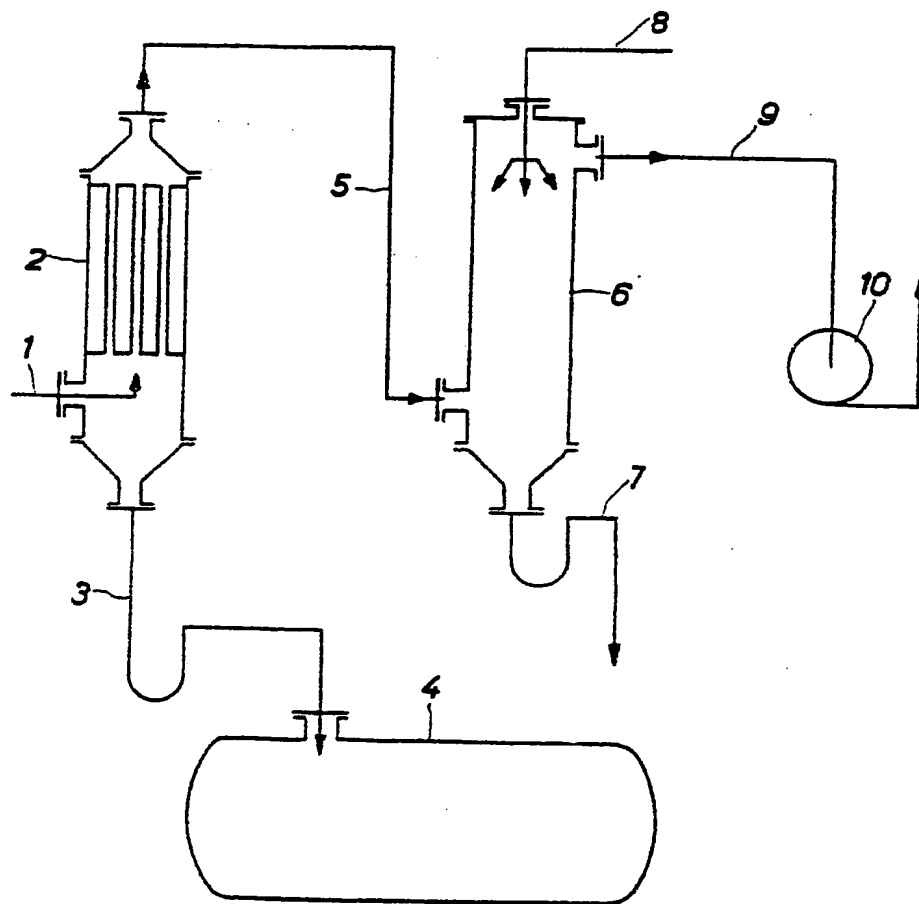


FIG. 2

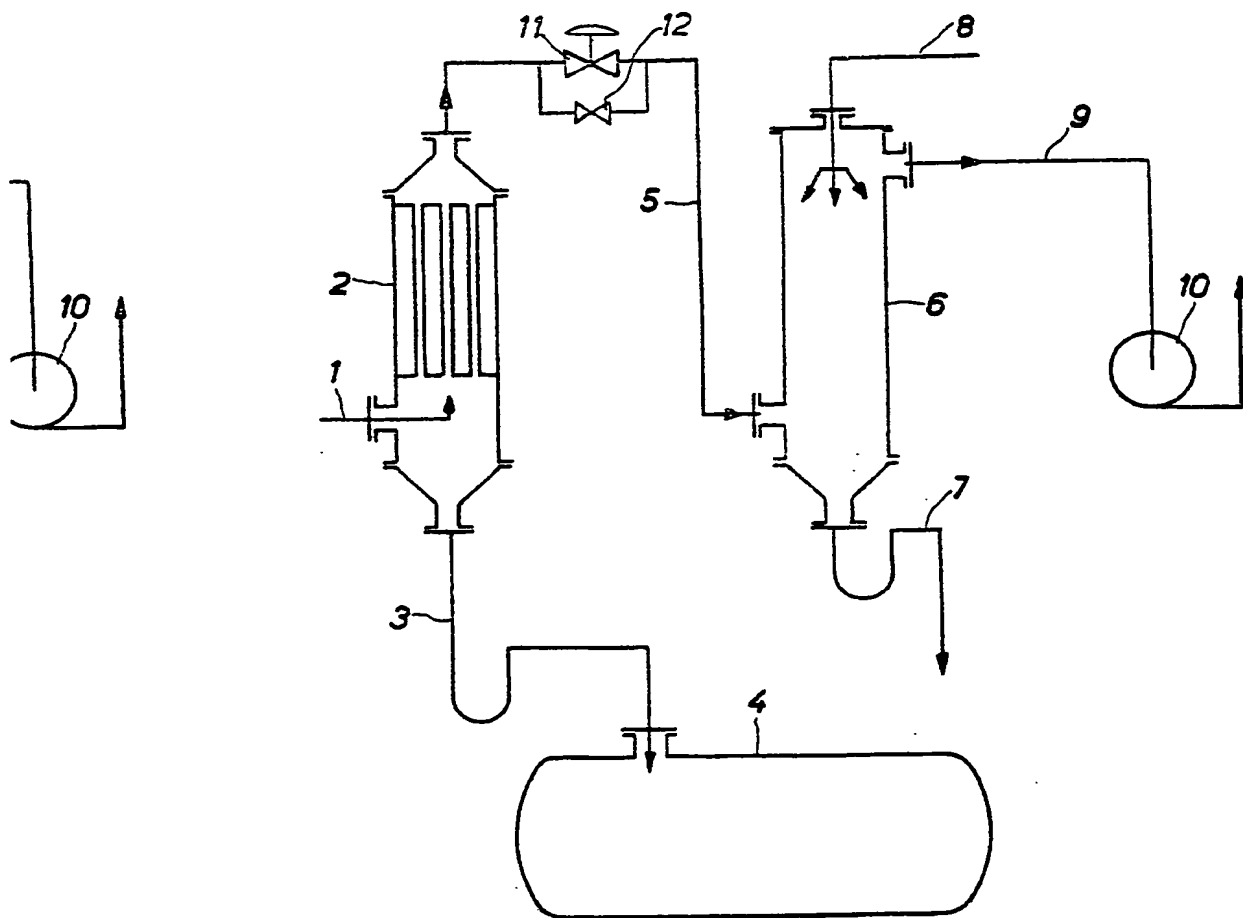


FIG.1

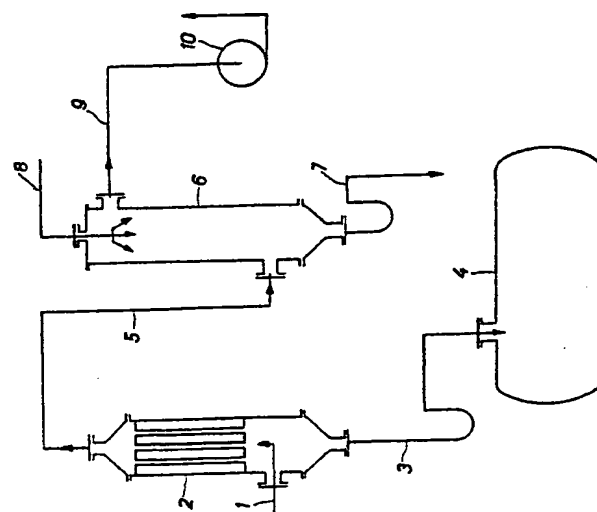
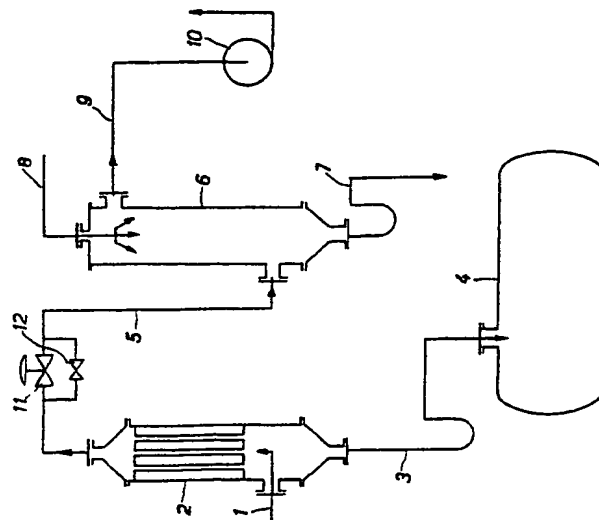


FIG.2



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